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Adsorption of reactive dye on chitosan in *air-lift* reactor

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Abstract This study was undertaken to identify factors exerting the strongest influence on the adsorption of dye. The maximum adsorption capacity (at the adopted operating conditions) was the main parameter used to evaluate the process. In addition, the feasible adsorption capacity of chitosan was evaluated. Breakthrough experiments were carried out in a circulating air-lift reactor at a constant concentration of reactive dye Black 8 (100 mg/dm^3). The tests studied different chitosan concentrations in the reactor and a range of flow intensities. The results of the breakthrough tests were compared by means of apparent mass transfer coefficients, determined by slopes at $C/C_0 = 1/2$. The adsorption capacity of chitosan was affected to the greatest extent by the flow rate of the medium to the reactor. In turn, the utilization of the maximum adsorption capacity of chitosan, at the assumed efficiency of dye removal, was determined by chitosan concentration in the reactor.

Keywords Adsorption · Chitosan · *Air-lift* reactor · Black 8

1 Introduction

Removal of dyes from waste waters poses severe difficulties since they often display a bacteriostatic and even bactericidal activity (McMullan et al. 2001; Lazaridis et al. 2003). Hence, the biological treatment results in either a lack or a slight change in the color of the post-dyeing sewage (Karcher et al. 2001; Anielak 1995).

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Wide acceptance of an adsorption process to address this problem depends on economics, which is mostly linked to the price of the adsorbent and the feasibility of its regeneration.

The adsorption process can be carried out in co-flow or counter-flow one-step or multi-step portion system. The rate of the process depends on the size of adsorbent molecules, i.e. the smaller the molecules, the higher the rate of the process. For this reason, it is advisable to use adsorbents in a powdery form (Anielak 2000). However, the removal of adsorbent from a purified solution still poses some difficulties.

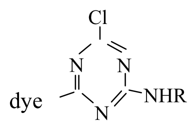
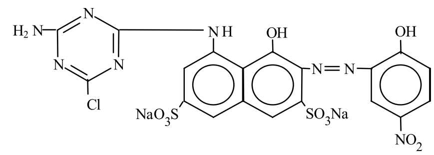
Adsorption can be conducted with the adsorbent in a moving-bed or fixed-bed. In adsorbers with stationary beds, the adsorbent with spent adsorption capacity may be periodically discharged for regeneration, whereas in adsorbers with moving (pulsing) beds the flowing solution is periodically stopped for exchange of the adsorbent.

Fixed-bed adsorbers can work as single adsorbers; or as two or more—connected in series; two or more connected in parallel; as well as four or more in a series-parallel system. Adsorption can also be carried out in stirred vessels, using powdered adsorbent. They can be applied in, among others, pharmaceutical industry, environmental protection, foodstuff production, and in a variety of economic sectors (Pohorecki 2002).

Depending on the method of supplying energy, reactors may be divided into three groups: mechanically-driven ones e.g. tanks with a stirrer and multi-step columns with stirrers; hydraulic-driven ones e.g. flux reactors, and pneumatically-driven ones e.g. pulsed-flow columns and *air-lift* reactors.

Due to their advantages, the latter, have been widely applied particularly in biotechnology and wastewater treatment (Dziubiński and Sowiński 2002). Advantages of the *air-lift* reactors include simple construct, a lack of mobile pieces, and a high efficiency of gas-liquid mass exchange,

Table 1 Characteristics of dye

Reactive group	Class	Structural formula	Reactive Black 8	Molecular mass g/mole
chlorotriazine	azo-aminochlorotriazine			656.5

whereas their drawbacks are poor mixing, limited use for media of low viscosity and foaming. Yet, those factors do not exert any significant effect on the efficiency of reactor work in the course of the adsorption process.

Our previous investigations have demonstrated feasibility of an *air-lift* reactor for dye removal in the adsorption process (Filipkowska 2004, 2005). Conventional reactors, in which gas is the factor enforcing the flow of liquid, are currently more extensively applied in the chemical industry, biotechnology and wastewater treatment. A rapid advance in biotechnology observed in recent years has resulted in a number of modifications of reactors used. Air-lift reactors are important among the reactors utilizing air as a carrier. They are convenient for running both chemical and biological processes. Their classification depends on the mode of recirculation—external or internal. Their typical trait is a very high circulation of liquid mass (Chisti and Moo-Young 1987; Pohorecki 2002).

This study was undertaken to assess the efficiency of Black 8 removal by adsorption using chitosan in an *air-lift* reactor. Tests measured the effect of adsorbate concentration and flow rate on the total adsorption capacity of the adsorbent. They also studied the residence time of the reactor as well as the utilization of the adsorption capacity at an assumed efficiency of dye removal.

2 Experimental procedures

2.1 Chitosan preparation and characteristics

Krill chitin originating from the Sea Fisheries Institute in Gdynia was used in the experiment. Its dry weight content accounted for 95.64% and ash content for 0.32%. An average size of a chitin flake used in the study was 314 × 184 μm × 92 μm.

Chitin was prepared following the method postulated by Stanley et al. (1975). Preparation of chitin involved: rinsing with distilled water, rinsing with hydrochloric acid in order to wash out calcium and magnesium ions, and next cooking with 70% potassium hydroxide. The degree of acetylation of so prepared chitosan accounted for 75%. The chemical structure of a chitosan molecule was presented in Fig. 1.

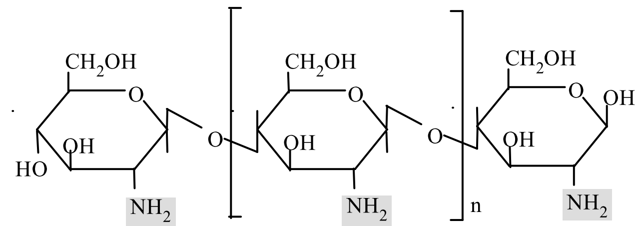


Fig. 1 Chemical structure of a chitosan molecule

2.2 Dye preparation

The chlorotriazine reactive dye Black 8 from "Boruta" SA Dye Plant in Zgierz was used in the experiment. Its chemical structure and characteristics were presented in Table 1.

2.3 Analysis of the efficiency of Black 8 adsorption onto chitosan in an *air-lift* reactor

The experiment was carried out in an *air-lift* reactor, the scheme of which is shown in Fig. 2. Use was made of a reactor of a circular section made of plexiglass, with a diameter of 0.06 m, height of 0.54 m, and active volume of 0.77 dm^3. In the lower part of the reactor, there were fixed truncated cone-shaped stub pipes allowing the inflow of air and dye solution. Inside the reactor, a 0.5 m long barrier was installed centrally. Near the effluent of the reactor there was a separator.

Analyses of Black 8 adsorption onto chitosan in the *air-lift* reactor were carried out at different inlet flow rates and chitosan concentrations in the reactor. The inlet dye concentration was constant and reached 100 mg/dm^3. The pH of the solution was maintained at a level of 5.0. Technological parameters of the research were presented in Table 2.

The analysis of Black 8 adsorption onto chitosan was carried out as follows: an appropriate concentration of chitosan was transferred into the reactor filled with water of pH 5.0. Aeration was turned on at a pressure of 0.15 MPa, in order to force chitosan movement in the reactor. Then, a solution with dye of a specified concentration (100 mg/dm^3) was dosed by means of a peristaltic pump at a rate of 0.77 dm^3/h, 1.54 dm^3/h and 3.1 dm^3/h, which corresponded to the flow

Table 2 Technological assumptions of RB8 adsorption analyses in the *air-lift* reactor

Parameter		Unit	Values
Flow rate	(q)	V/h (dm^3/h)	1, 2, 3
Chitosan concentration	(m)	g dry weight/dm^3	1, 2, 5, 10
Inlet dye concentration	(C_0)	mg/dm^3	100

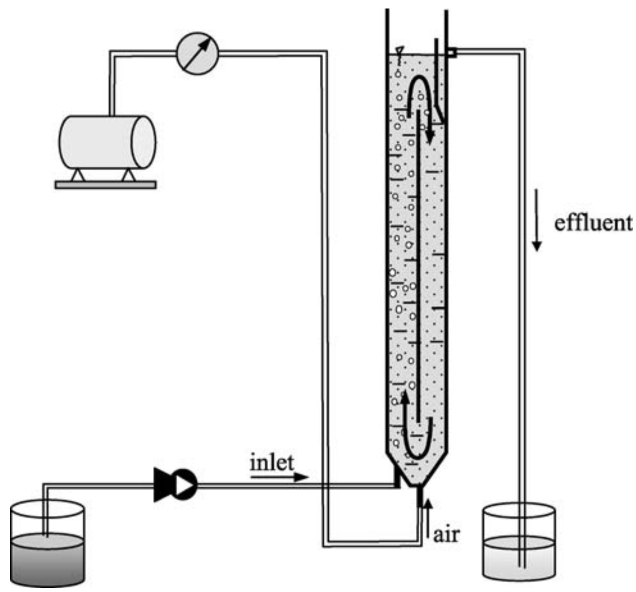


Fig. 2 Scheme of the *air-lift* reactor

rate of 1 V/h, 2 V/h and 3 V/h (where V denotes reactor volume).

A total of 12 experimental series were carried out that differed in chitosan concentration in the reactor and flow rate.

In each series, analyses were carried out as long as the concentration of dye in the effluent was equal to its initial inlet concentration. For each flow rate, control series were performed without chitosan. Results obtained from control and experimental series enabled determining the adsorption capacity of chitosan under dynamic conditions.

3 Analytical methods

pH was measured with the use of an HI 123 pH-meter, chitosan concentration was determined as chitosan dry weight according to the methodology described by Hermanowicz et al. (1999). Deacetylation degree of chitosan was analyzed according to Roberts (1992).

3.1 Determination of dye concentration

Dye Reactive Black 8 was assigned a visual wavelength ($\lambda = 587$) at which absorbance was measured in order to plot a standardization curve and compute a conversion coefficient. Dye concentration was measured spectrophotometrically using a UV-VIS Spectrophotometer SP 3000 apparatus.

3.2 Determination of dye concentration in the *air-lift* reactor

The efficiency of the process was calculated by monitoring the decolourisation process. At regular time intervals (10 min) several samples were taken which were then centrifuged (15.000 rpm) for 10 min. The residual dye concentration was measured spectrophotometrically at 587 nm.

4 Results and discussion

The quantity of dye adsorbed onto chitosan Q_r under flow conditions was calculated from (1):

$$Q_r(t) = \frac{\sum_{t=1}^{n}((C_t - C_{kt}) \cdot q \cdot t)}{V \cdot m} \tag{1}$$

$Q_r(t)$ quantity of dye adsorbed onto chitosan in time t (mg/g dry weight); C_t mean concentration of dye in effluent in time t in the control sample (mg/dm^3); C_{kt} mean concentration of dye in effluent outlet in time t (samples with chitosan) (mg/dm^3); q flow rate of a dye solution to the reactor (dm^3/h); t adsorption time (h); V reactor volume (dm^3); m chitosan concentration in the reactor (g dry weight/dm^3).

The experimental results yielded the adsorption capacity of chitosan in any time, t, of the experiment. Its final value was the maximal adsorption capacity of chitosan.

Figure 3 shows the total adsorption capacity of chitosan obtained at various concentrations of chitosan in the *air-lift* reactor for the three flow rates examined. In all experimental series, dye concentration was constant and accounted for 100 mg/dm^3.

Investigations demonstrated that, under dynamic conditions, the adsorption capacity of chitosan depended on the flow rate. At the flow rate of 1 V/h, irrespective of the content of adsorbent in the reactor, the adsorption capacity ranged from 4551 to 4298 mg/g dry weight. At a twofold higher flow rate, the quantity of dye adsorbed was lower and ranged from 2573 to 2210 mg/g dry weight on average. Increasing the flow rate to 3 V/h resulted in a decrease in the adsorption capacity—from 1842 to 1421 mg/g dry weight.

Table 3 Working time of the reactor depending on the assumed dye concentration in effluent

Chitosan concentration	Working time of the reactor (min)											
	0.77 dm^3/h (*V*/h)				1.54 dm^3/h (2 *V*/h)				2.31 dm^3/h (3 *V*/h)			
	100%	99%	95%	90%	100%	99%	95%	90%	100%	99%	95%	90%
1 g dry weight/dm^3	30	75	120	160	0	5	55	80	0	5	20	35
2 g dry weight/dm^3	240	250	295	315	170	185	225	245	125	130	140	155
5 g dry weight/dm^3	1025	1030	1045	1080	505	535	590	610	360	380	400	420
10 g dry weight/dm^3	2070	2075	2080	2105	1020	1022	1025	1045	605	610	620	630

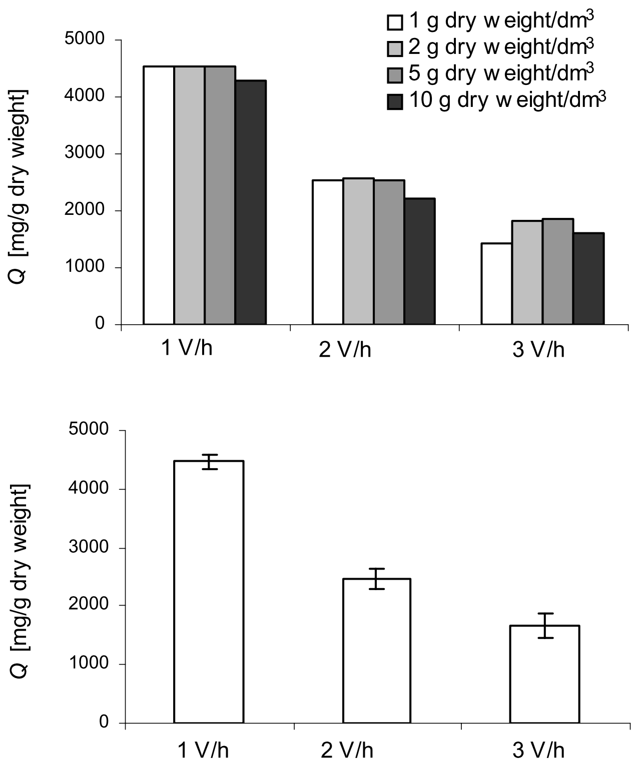


Fig. 3 Total adsorption capacity of chitosan depending on flow rate of dye and chitosan concentration in the *air-lift* reactor

Analogous results were obtained by Sag et al. (2003) who investigated adsorption of metal ions onto chitin. He demonstrated that the content of metal ions adsorbed onto chitin was decreasing with an increasing flow rate. Similar conclusions were drawn in a paper by Sag and Aktay (2001) who analyzed the efficiency of Cr (VI) ions removal onto chitin in reactor columns under dynamic conditions. The maximum capacity of chitin filling the reactor columns was expected to increase along with an increasing flow rate due to high loading of the bed with metal ions. Yet, a shorter contact of metal with the sorbent reduced the quantity of metal adsorbed on the surface of chitin. The load of metal adsorbed on the surface of chitin was observed to decrease with an increasing flow rate. Similar findings were reported too by Padmesh et al. (2006). They investigated the influence of flow rate on biosorption of AB15 by keeping initial dye concentration (100 mg/dm^3) and varying the flow rate. The results obtained demonstrated that earlier breakthrough time appeared for highest flow rate, resulting in low uptake of dye. This behavior may be due to insufficient time for the solute inside the column and the diffusion limitations of the solute into the pores of the sorbent at higher flow rates.

Table 3 summarizes the working time of the reactor depending on the assumed content of dye in the effluent. Four efficiencies of dye removal were assumed in the study: 100% (a lack of dye in the effluent), 99%, 95% and 90% which corresponded to 1, 5 and 10 mg/dm^3 of dye in the effluent, respectively.

The longest working time of the reactor—over 2000 min—was noted in experimental series with the lowest flow rate (1 *V*/h) and with the highest concentration of adsorbent in the reactor (10 mg/g dry weight). At the lowest chitosan concentration—1 g dry weight/dm^3, the working time of the reactor was the shortest, at the assumed efficiency of dye removal. Higher flow rates (2 *V*/h and 3 *V*/h) did not allow the complete removal of dye. An increased concentration of chitosan in the reactor distinctly affected elongation of the working time. Consistent findings were reported by Lalov et al. (2000). They investigated the treatment process of vinasse-containing water through ionic exchange onto chitosan (anion exchanger) and proved that various concentrations of chitosan affected the working time of the reactor. At chitosan concentration of 10 and 15 g/dm^3 the assumed initial concentration was reached after 90 min and the process was completed after 300 min, whereas at lower concentrations of chitosan the process terminated after 180 min.

Benguella and Benaissa (2001), based on investigations into the possibility of removing cadmium ions onto chitin, also reported that the concentration of chitin affected the time of the adsorption process necessary to reach the state of dynamic equilibrium.

Benguella et al. claimed that at the lowest analyzed concentration of chitin—1 g/dm^3, the time needed for reaching equilibrium in the reactor was 1.5 h, whereas at chitin concentration of 6 g/dm^3 that time was elongated to 13 h.

Table 4 Utilization of the adsorption capacity of chitosan depending on the assumed dye concentration in effluent

Chitosan concentration	Q/Q_{max} (%)											
	0.77dm^3/h (V/h)				1.54 dm^3/h (2 V/h)				2.31 dm^3/h (3 V/h)			
	100%	99%	95%	90%	100%	99%	95%	90%	100%	99%	95%	90%
1 g dry weight/dm^3	2.1	12.6	27.3	41.3	0.0	0.5	21.9	37.8	0.0	0.6	8.7	21.9
2 g dry weight/dm^3	50.9	53.7	66.3	71.6	50.7	56.3	71.1	78.0	57.3	60.0	65.3	72.9
5 g dry weight/dm^3	84.9	85.3	86.6	89.5	75.0	79.7	88.2	91.1	73.8	77.0	82.3	86.3
10 g dry weight/dm^3	93.6	93.9	94.1	95.1	89.7	90.0	90.1	91.8	73.1	73.7	74.9	76.2

In addition, those authors proved that the mass of chitin affected the efficiency of adsorption process since the increasing concentration of chitin was accompanied by an increasing quantity of metal removed from the solution. They explained this by the fact that a higher concentration of chitin created a greater surface area of contact with cadmium ions.

The latter has been confirmed by results of the reported study. At a lower flow rate, the time of contact between molecules of a dye and a sorbent was longer than at the higher flow rates. The possibility of adsorbing the dye onto sorbent and, consequently, the total load of removed adsorbate was thus greater.

The results obtained for the working time of the reactor at an assumed efficiency of dye removal were demonstrated to affect the adsorption capacity.

Table 4 presents Q/Q_{max} values depending on the adopted technological conditions. The Q value indicates the quantity of dye adsorbed onto chitosan at a corresponding assumed efficiency of dye removal reaching 100%, 99%, 95% and 90%, whereas the Q_{max} value denotes the total quantity of dye adsorbed onto chitosan when $C = C_0$.

The research demonstrated that at the constant inlet concentration of dye the utilization of adsorption capacity of chitosan depended to a greater extent on the concentration of adsorbent in the reactor than on the inlet concentration, as shown in the case of the total adsorption capacity. At the lowest tested concentration of chitosan, i.e. 1 g dry weight/dm^3, a very low utilization of adsorbent was demonstrated at the assumed efficiency of dye removal even at the flow rate of 1 V/h. Analogous results were obtained during adsorption in the *air-lift* reactor onto chitin (Filipkowska 2005), where a low concentration of chitin had also a negative effect on the quantity of dye adsorbed and utilization of adsorbent. In all other experimental series, utilization of the adsorption capacity of chitosan, at the assumed efficiency of dye removal, was higher than 50%. The best results were reported at the highest concentration of chitosan reaching 10 g dry weight/dm^3, when at flow rates of 1 V/h and 2 V/h the utilization of the adsorption capacity of chitosan reached 90% even at the highest assumed efficiency of dye removal (100%).

Table 5 Values of tangent coefficients a (tangent inclination) and b

Chitosan concentration	Constants					
	1 V/h		2 V/h		3 V/h	
	a	b	a	b	a	b
1 g dry weight/dm^3	0.345	−0.839	0.238	−0.449	0.199	−0.188
2 g dry weight/dm^3	0.453	−1.964	0.289	−1.76	0.221	−1.19
5 g dry weight/dm^3	0.486	−6.872	0.432	−6.85	0.234	−3.82
10 g dry weight/dm^3	0.507	−13.780	0.499	−8.149	0.354	−8.612

The results obtained and breakthrough curves plotted enabled stating that the selection of parameters of reactor work, including: adsorbent concentration, adsorbate concentration and flow rate of medium through the reactor, has a significant effect on the course of adsorption curves. The plots of C/C_0 versus volume are given in Fig. 4.

For most adsorption operations in water and wastewater treatment, breakthrough curves exhibit a characteristic "S" shape, but with varying degree of steepness and position of the breakpoint. For all experimental series, tangents were plotted at $C/C_0 = 1/2$. Values of tangent coefficients a (tangent inclination) and b were presented in Table 5.

A distinct tendency was observed for tangent inclination (a) depending on the parameters of reactor work. The highest values of constant a were noted in the series when the highest adsorption capacity of chitosan was reached simultaneously at the assumed efficiency of dye removal. Values of the constants given in Table 5 enable stating that the course of the adsorption process, at the comparable adsorption capacity obtained, is more favorable once the tangent to the breakthrough curve in point $C/C_0 = 1/2$ is more steep, i.e. when the coefficient a is higher.

The experimental results enable concluding that the adsorption capacity of chitosan is determined by the flow rate and that the load of dye removed decreases with an increasing flow rate. Still, while adjusting the appropriate flow rate caution should be paid to factors affecting a decrease in adsorption process effectiveness under dynamic conditions.

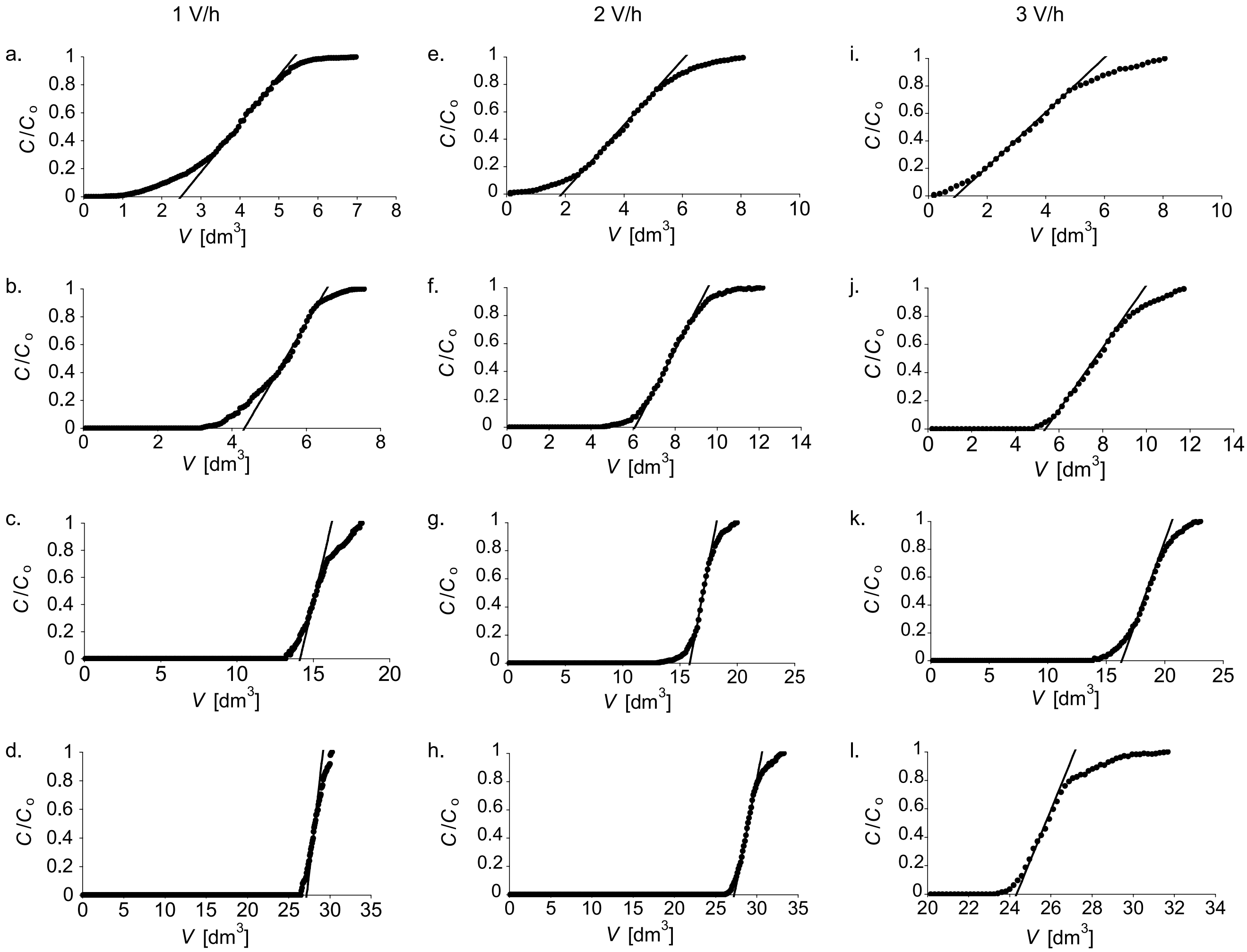


Fig. 4 Breakthrough curves and tangent of Black 8 at a flow rate of 1 V/h: (**a**) 1 g dry weight/dm^3, (**b**) 2 g dry weight/dm^3, (**c**) 5 g dry weight/dm^3 and (**d**) 10 g dry weight/dm^3, at a flow rate of 2 V/h (**e**) 1 g dry weight/dm^3, (**f**) 2 g dry weight/dm^3, (**g**) 5 g dry weight/dm^3 and (**h**) 10 g dry weight/dm^3, and at a flow rate of 3 V/h (**i**) 1 g dry weight/dm^3, (**j**) 2 g dry weight/dm^3, (**k**) 5 g dry weight/dm^3 and (**l**) 10 g dry weight/dm^3

5 Conclusions

This research was undertaken to determine the efficiency of Black 8 removal from aqueous solutions at pH 5.0 by adsorption onto chitosan. The analyses carried out in the study enabled determining the impact of chitosan concentration and flow rate on the course of the adsorption process as well as determining the optimal parameters and factors inhibiting work of the *air-lift* circulating reactor.

The results obtained in the study indicate that:

1. At the assumed efficiency of dye removal, the adsorption capacity of chitosan is determined by flow rate. With the increasing flow rate, the total adsorption capacity of chitosan was observed to decrease. The greatest load of adsorbed Black 8 was obtained at the lowest flow rate. Increasing the flow rate to 2 V/h (1.54 dm^3/h) resulted in a decrease of the total load of adsorbed Black 8 by 56% on average, as compared to 1 V/h (0.77 dm^3/h). The subsequent increase of the flow rate to 3 V/h (2.31 dm^3/h) decreased the total load of Black 8 adsorbed by another 30% in respect to the load obtained at the flow rate of 2 V/h and by 62% in respect to that obtained at 1 V/h.
2. Flow rate affected the working time of the reactor. With an increasing flow rate, the time required for complete breakthrough ($C = C_0$) decreased. The shortest working time of the reactor was obtained at the highest flow rate. Also chitosan concentration in the reactor was found to influence the working time of the reactor; i.e. the higher the concentration of chitosan the longer the adsorption process. It appeared to be the longest at chitosan concentration equal to 10 g dry weight/dm^3.
3. The concentration of chitosan did not affect the total quantity of dye adsorbed at the constant flow rate. Values of the adsorbed load at the four chitosan concentra-

tions examined were comparable for the three flow rates applied. In turn, chitosan concentration had a significant effect on the utilization of adsorption capacity at the assumed efficiency of dye removal, i.e. the higher the concentration of chitosan in the reactor the higher the quantity of dye adsorbed at the assumed dye concentration in the effluent.

Nomenclature

- $Q_r(t)$ quantity of dye adsorbed onto chitosan in time t (mg/g dry weight)
- C_t mean concentration of dye in effluent in time t in the control sample (mg/dm^3)
- C_{kt} mean concentration of dye in effluent outlet in time t (samples with chitosan) (mg /dm^3)
- q flow rate of a dye solution to the reactor (dm^3/h)
- t adsorption time (h)
- V reactor volume (dm^3)
- m chitosan concentration in the reactor (g dry weight/dm^3)
- C_0 initial concentration of dye (mg/dm^3)
- Q value indicates the quantity of dye adsorbed onto chitosan at a corresponding assumed efficiency of dye removal reaching 100%, 99%, 95% and 90% (mg/g dry weight)
- $Q_{\max}$ value denotes the total quantity of dye adsorbed onto chitosan when $C_t = C_0$ (mg/g dry weight)
- $\lambda_{\max}$ maximum wavelength (nm)

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